RPT-174

Data Summary Report for Operable Unit 3-13, Group 3, Other Surface Soils, Remediation Site CPP-97 Confirmation Sampling

February 2006

Idaho Cleanup Project

The Idaho Cleanup Project is operated for the
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February 2006

Idaho Cleanup Project
Operable Unit 3-13, Group 3
Idaho Falls, Idaho 83415

Prepared for the
U.S. Department of Energy
Assistant Secretary for Environmental Management
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14516

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Data Summary Report for Operable Unit 3-13, Group 3, New Soil Release Sites, Remediation Site CPP-97 Confirmation Sampling

1. PURPOSE

This report presents confirmation sampling data obtained subsequent to removing two piles of contaminated soil at the Idaho Nuclear Technology Engineering Center remediation site, CPP-97, at the Idaho National Laboratory.

2. SCOPE

The confirmation sampling data are summarized in the body of this report. Appendix A contains the limitations and validation report from which the data summary was extracted.

3. BACKGROUND

Site CPP-97 consisted of two tarp-covered soil stockpiles that originated from the Tank Farm Upgrade Project. One pile contained approximately 1,093 m^3 (1,430 yd^3) of radionuclide-contaminated soil. Radiation measurements at the time of generation ranged between 0 and 3 mR/hour. The second soil stockpile contained approximately 53 m^3 (70 yd^3) of radionuclide-contaminated soil, with 3–50 mR/hour radiation readings. These soil stockpiles were included in Group 3, Other Surface Soils, for remediation and disposal at the Idaho Comprehensive Environmental Response, Conservation, and Liability Act Disposal Facility.

The Idaho Nuclear Technology Engineering Center Record of Decision (DOE-ID 1999) identified site CPP-97 as needing remediation and established remediation goals for the contaminants of concern (COCs) (i.e., Cs-137 and Sr-90). The site was subsequently remediated in accordance with the Remedial Design/Remedial Action Work Plan (DOE-ID 2004a). Confirmation sampling was then conducted over the site to verify that the remediation goals were met in accordance with the Field Sampling Plan (FSP) (DOE-ID 2004b). The FSP also stated that if the residual concentrations of Cs-137 and Sr-90 were below the remediation goals, then no further remedial action would be required for the soil, and remediation would be considered complete at this site. Table 1 summarizes contaminant information and the associated remediation goals for site CPP-97 COCs. The FSP further identified the number of samples to be collected, sampling locations, method of sample collection, handling and packaging procedures, shipping procedures, and methods of analyses. All activities pertaining to the confirmation sampling and reported in this document were conducted in accordance with the FSP.

Table 1. Soil risk-based remediation goals.

Site	Description	Contaminants of Concern	Remediation Goal[a]
CPP-97	Tank Farm soil stockpile: two tarp-covered stockpiles and contaminated surface soil	Cs-137	23 pCi/g, or the natural logarithm transform: 3.14 ln(pCi/g)
		Sr-90	223 pCi/g

a. Risk-based remediation goals developed for the residential scenario.

4. SITE CPP-97 SOIL SAMPLING AND ANALYSES

Eight soil samples were collected from site CPP-97 and shipped to a contract analytical laboratory. Figure 1 shows confirmation sampling locations. The contract analytical laboratory analyzed the soil samples using gamma spectroscopy methods for Cs-137 and gas flow proportional counting methods for total strontium (which inherently includes Sr-90), as specified in the FSP.

5. DATA SUMMARY

The contract analytical laboratory data sheets for the analyses are included as an attachment in Appendix A. The limitations and validation report (see Appendix A) qualifies the data, which are summarized in Table 2, for the Operable Unit 3-13 COCs only. Cesium-137 and Sr-90 were the only COCs identified for Operable Unit 3-13 site CPP-97; however, the analytical method used to detect Cs-137 is also sensitive to several other radionuclides. These radionuclides are listed in Appendix A; however, they are not included in Table 2 because these radionuclides are not identified in the Record of Decision as COCs. Table 3 presents the concentrations of Cs-137 in the eight samples, while Table 4 presents total strontium in the eight samples. Sample E0730500101RH was split into two samples for quality control purposes, with the second sample assigned the number E0730500102RH. Both values are shown in the tables; however, the maximum value between the two samples was used to calculate statistical information.

The Cs-137 data were unqualified, statistically positive in the 95% confidence interval, and above the minimum detectable activity. Consequently, the data were considered unbiased and representative of the samples.

All of the total strontium data were qualified, but not rejected. Consequently, the "J"-qualified data were used to assess the completeness of remediation for the site. The qualification arose because the Laboratory Control Samples (LCSs) were biased low. The biased condition required the field sample data to be qualified and used with discretion, but did not necessarily ignore them. The following is excerpted from the attached limitations and validation report (see Appendix A).

> Laboratory Control Samples (LCSs) are used to assess the bias and precision of the analytical process independent of the field samples. The LCS results (percent recovery) are also used to indicate whether the laboratory's radiochemical procedure is capable of recovering the radionuclide of interest.
>
> Total Sr LCS recovery was 73%, which is less than the 75–125% acceptance criteria. . .(so). . .all of the statistically positive Total Sr results were qualified "J."

The bias implies that the field sample data are 27% lower than the actual values for total strontium that would have been obtained had the LCS recovery been 100%. This limitation (bias) in the data is insignificant because the field sample values are approximately two orders of magnitude lower than the remediation goal. Based on this information, the sample data results are adequate for use in determining whether the Operable Unit 3-13 soil risk-based remediation goals for strontium have been achieved.

The Shapiro-Wilk test was used to test the sampling data for normality. The Cs-137 results were significantly non-normal ($p = 0.026$) but did not fail for log-normality ($p = 0.96$). Natural log-transformed values were used to calculate the upper confidence limit (UCL) and to compare to the natural log-transformed remediation goal, as specified in Section 3.1.5 of the FSP.

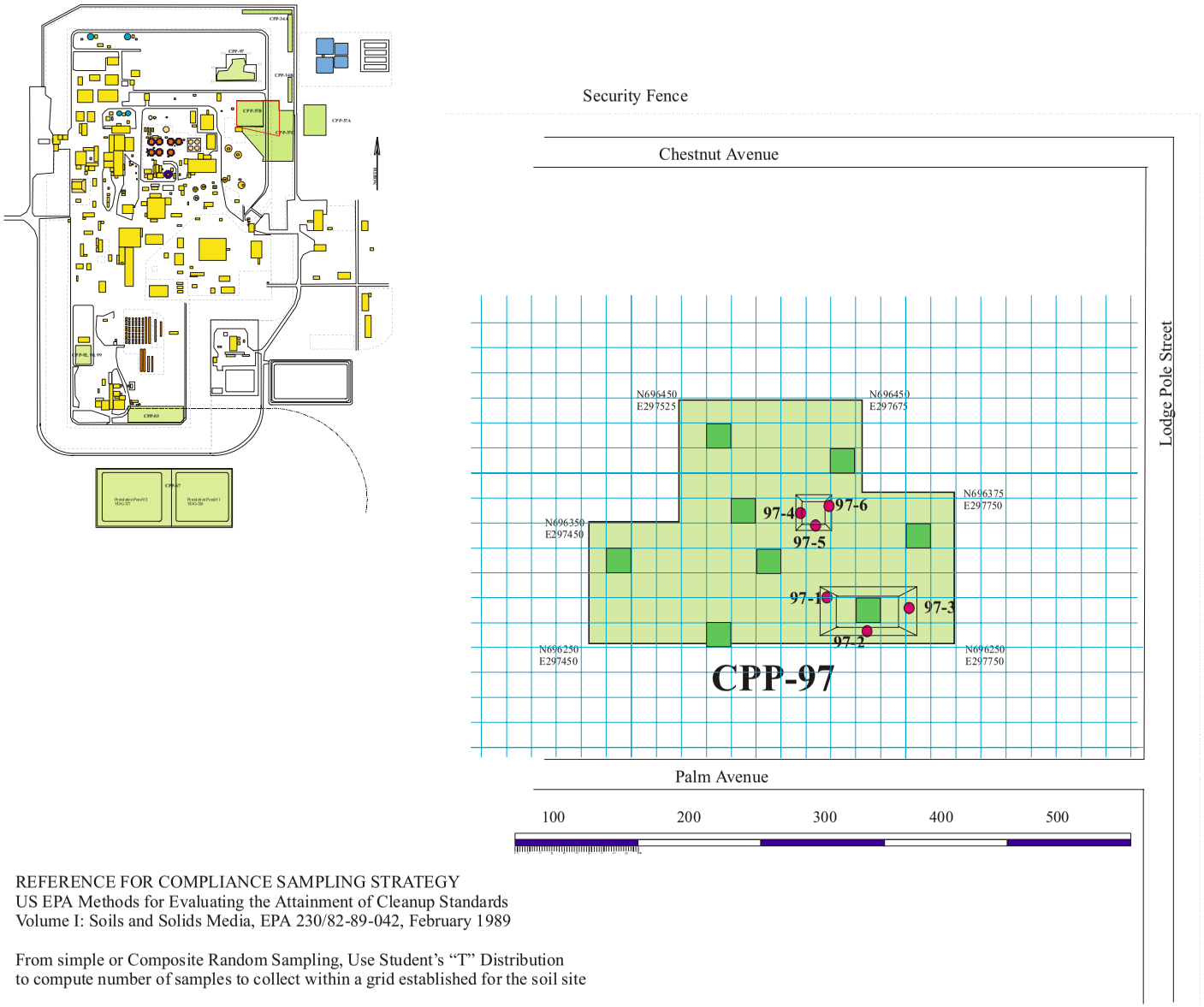

Figure 1. Confirmation sampling locations for site CPP-97.

Table 2. Qualification assignment for the confirmation samples collected on site CPP-97.

Sample No.	Cs-137	Total Strontium
E0730500101RH	—	U
E0730500102RH	—	J
E0730500201RH	—	J
E0730500301RH	—	J
E0730500401RH	—	J
E0730500501RH	—	J
E0730500601RH	—	J
E0730500701RH	—	J
E0730500801RH	—	J

— = The analysis was performed; radioactivity was detected (statistically positive at the 95% confidence interval) and is above the minimum detectable activity. The radionuclide is considered to be present in the sample, and the results are not qualified.

U = The analysis was performed; no radioactivity was detected (not statistically positive at the 95% confidence interval) or the result was less than the minimum detectable activity. The radionuclide is not considered to be present in the sample.

J = The analysis was performed, and radioactivity was detected. The result is statistically positive at the 95% confidence interval and is greater than the minimum detectable activity. The result is questionable. The radionuclide is considered to be in the sample; however, the result may not be accurate.

Table 3. Cesium-137 concentrations in the eight confirmation samples.

Sample No.	Cs-137 (pCi/g)	Natural Log-Transformed Activities Used to Calculate Statistical Information for Cs-137 [ln(pCi/g)]	Total Uncertainty (±1 sigma) (pCi/g)	Minimum Detectable Activity (pCi/g)
E0730500101RH	1.47E-01	—	0.041	0.083
E0730500102RH	3.23E+00	1.17	0.280	0.090
E0730500201RH	1.03E+00	0.03	0.097	0.083
E0730500301RH	7.22E-01	-0.33	0.087	0.081
E0730500401RH	2.37E+00	0.86	0.160	0.070
E0730500501RH	4.56E-01	-0.78	0.059	0.090
E0730500601RH	9.92E+00	2.29	0.780	0.080
E0730500701RH	5.26E+00	1.66	0.410	0.080
E0730500801RH	9.47E-01	-0.05	0.094	0.087
Average	—	0.61	—	—
Standard deviation	—	1.06	—	—
95% confidence interval width	—	0.74	—	—
95% upper confidence limit for the mean	—	1.34	—	—

Table 4. Strontium concentrations (for all radioisotopes) in the eight confirmation samples.

Sample No.	Total Strontium (pCi/g)	Values Used to Calculate Statistical Information (pCi/g)	Total Uncertainty (±1 sigma) (pCi/g)	Minimum Detectable Limit (pCi/g)
E0730500101RH	1.50E-02	—	5.50E-02	1.90E-01
E0730500102RH	3.19E+00	3.19	-1.90E-01	1.20E-01
E0730500201RH	1.87E-01	0.19	-4.20E-02	1.20E-01
E0730500301RH	6.08E+00	6.08	-3.30E-01	1.30E-01
E0730500401RH	2.25E+00	2.25	-1.60E-01	1.90E-01
E0730500501RH	2.21E+00	2.21	-1.30E-01	9.00E-02
E0730500601RH	5.80E-01	0.58	-8.90E-02	2.30E-01
E0730500701RH	8.44E+00	8.44	-4.50E-01	1.10E-01
E0730500801RH	2.85E+00	2.85	-1.70E-01	1.30E-01
Average	—	3.22	—	—
Standard deviation	—	2.77	—	—
95% confidence interval width	—	1.92	—	—
95% upper confidence limit for the mean	—	5.14	—	—

The Sr-90 results did not differ significantly from normal ($p = 0.28$), so these data were used without transformation.

The final residual concentration for Cs-137 over site CPP-97 is calculated by averaging the log-transformed values from the eight confirmation samples. The final result is 0.61 ln(pCi/g) plus or minus the 95% confidence interval width of 0.74 ln(pCi/g), or the 95% UCL for the natural log-transformed mean is less than or equal to 1.34 ln(pCi/g). The natural log-transformed remediation goal is ln(23 pCi/g) = 3.14 ln(pCi/g).

The final residual concentration for Sr-90 (as calculated for total strontium) over site CPP-97 is calculated by averaging the values from the eight confirmation samples. The final result is 3.22 pCi/g plus or minus the 95% confidence interval width of 1.92 pCi/g, or the 95% UCL for the mean is less than or equal to 5.14 pCi/g.

The 95% confidence interval width was calculated using Equation (1):

$$\text{Confidence interval} = \bar{x} \pm 1.96(s/\sqrt{n}) \quad (1)$$

where:

$\bar{x}$ = the mean

s = standard deviation

$\sqrt{n}$ = square root of n

n = number of samples.

6. CONCLUSION

Confirmation samples collected at site CPP-97 after remediation showed the natural log-transformed average residual concentration for Cs-137 to be 0.61 ± 0.74 ln(pCi/g). This value is substantially lower than the natural log-transformed remediation goal of 3.14 ln(pCi/g).

Confirmation samples collected at site CPP-97 after remediation showed the average residual concentration for strontium (all radioisotopes) to be 3.22 ± 1.92 pCi/g. This value is substantially lower than the remediation goal of 223 pCi/g.

Based on the above results, remediation of site CPP-97 is complete.

7. REFERENCES

DOE-ID, 1999, *Final Record of Decision (ROD) Idaho Nuclear Technology and Engineering Center (INTEC), Operable Unit (OU) 3-13*, DOE/ID-10660, Rev. 0, U.S. Department of Energy Idaho Operations Office, September 1999.

DOE-ID, 2004a, *Operable Unit 3-13, Group 3, Other Surface Soils Remediation Sets 1-3 (Phase I), Remedial Design/Remedial Action Work Plan*, DOE/ID-11089, Rev. 0, U.S. Department of Energy Idaho Operations Office, February 2004.

DOE-ID, 2004b, *Operable Unit 3-13, Group 3, Other Surface Soils Remediation Sets 1–3 (Phase I) Field Sampling Plan*, DOE/ID-11091, Rev. 0, U.S. Department of Energy Idaho Operations Office, February 2004.

Appendix A

Radioanalytical Data Limitations and Validation Report for the Radiological Analyses of Samples Collected at the INL in Support of the ESP-073-05, CPP-97 Field Sampling

Appendix A

Radioanalytical Data Limitations and Validation Report for the Radiological Analyses of Samples Collected at the INL in Support of the ESP-073-05, CPP-97 Field Sampling

Idaho Cleanup Project

CH2M•WG IDAHO, LLC

INTEROFFICE MEMORANDUM

Date:	January 11, 2006		
To:	Donna R. Kirchner	MS 3960	6-9873
From:	Susan Shinn SS	MS 3960	6-4898
Subject:	TRANSMITTAL OF THE RADIOLOGICAL LIMITATIONS AND VALIDATION (L&V) REPORT FOR THE ESP-073-05, CPP-97 FIELD SAMPLING, SDG # E0730500101RH		

Attached is the Limitation and Validation (L&V) report for the radiological analyses in support of the ESP-073-05, CPP-97 Field Sampling project, Sample Delivery Group (SDG) # E0730500101RH. Severn Trent Laboratories, Inc. (located in St. Louis, MO) analyzed nine (9) solid samples for total strontium and gamma-emitting radionuclide activity.

Portage Environmental, Inc. has validated the data package to analytical method data validation Level "A" as described in Guide Document (GDE)-7003, "Levels of Analytical Method Data Validation" and in accordance with GDE-205, "Radioanalytical Data Validation." I have reviewed the L&V report for correctness, completeness, consistency, and contractual compliance.

The ^{110m}Ag result for sample E0730500301RH, the ^{155}Eu result for sample E0730500401RH, the ^{235}U results for samples E0730500301RH, E0730500501RH, E0730500601RH and E0730500801RH were originally left unqualified. The sample results were statistically positive even at the 3-sigma level however, the results were below their respective MDAs. The sample results were re-qualified with "J" validation flags. The radionuclide is considered to be present in the samples; however, the results may not be an accurate representation of the amount of activity actually present in the samples.

Until such time as it becomes available through the applicable document control system, an electronic copy of the subject L&V report is available at:

http://icpweb/sampana/pdfApplication/IEDMS_PDF/Radiological/STL-STLOUIS/E0730500101RH/PD0397_E0730500101RH_SAMR1.pdf

For all data contained in the L&V report, the final decision of data usability is left to the discretion of the project manager. If you have any questions concerning these data or the L&V report, please contact me by telephone at 526-4898 or by e-mail at shins@inel.gov.

ss

Attachment

cc: Lee Davison, MS 5310
Donna Johnson, MS 3960/r/ARDC Files/r/Field Data Files
Joseph A. Landis, MS 5310
Kathy Otter, MS 3960
Randy S. Rice, MS 7110
Susan Shinn Letter File (SOS-TL012-06)

Uniform File Code: 7101
Disposition Authority: ENV5-c-3
Retention Schedule: Destroy in 75 years.

NOTE: Original disposition authority, retention schedule, and Uniform Filing Code applied by the sender may not be appropriate for all recipients. Make adjustments as needed.

RADIOANALYTICAL DATA LIMITATIONS AND VALIDATION REPORT

For the

RADIOLOGICAL ANALYSES OF

SAMPLES COLLECTED AT THE INL

In support of the

ESP-073-05, CPP-97 Field Sampling

Report Number:
CWI-PD0397-12-05

Analysis Types:
Gamma Isotopes
Total Sr

SDG No.
E0730500101RH

Prepared for: CH2M*WG Idaho, LLC (CWI)
P.O. Box 1625
Idaho Falls, ID 83415-3960

Prepared by: James Britton
Portage Environmental, Inc.
103 N. Main St.
Butte, MT 59701

Validator Signature: _____ Date: 12-22-05

1. PROJECT SCOPE/DESCRIPTION

A.	Project Name:	**ESP-073-05, CPP-97 Field Sampling**			
B.	L&V Report No.:	CWI-PD0397-12-05	I.	TOS Numbers:	ER-TOS-A2703
C.	SDG No.:	E0730500101RH			ER-SOW-394
D.	Number of Samples:	9	J.	Lab Name & Location:	STL, St. Louis, MO
E.	Sample type/matrix:	9, soil	K.	LTI Number:	F5K160391
F.	Analysis Type:	Gamma isotopes. Total Sr	L.	Validator Affiliation:	Portage
G.	Reporting Level:	Standard + Raw Data	M.	Validator:	Jim Britton
H.	Validation Level:	A	N.	Completion Date:	12/20/05

2. SAMPLE IDENTIFICATION

Field Sample Identification Number	Laboratory Identification Number	Sample Matrix
E0730500101RH	F5K160391-001	soil
E0730500102RH	F5K160391-002	soil
E0730500201RH	F5K160391-003	soil
E0730500301RH	F5K160391-004	soil
E0730500401RH	F5K160391-005	soil
E0730500501RH	F5K160391-006	soil
E0730500601RH	F5K160391-007	soil
E0730500701RH	F5K160391-008	soil
E0730500801RH	F5K160391-009	soil

3. ANALYSES AND METHODS

Analysis Type	Preparation/Analysis Method
Gamma Isotopes	DOE GA-01-R Mod.
Total Sr	DOE SR-03-RC Mod.

4. CONTRACT AND TECHNICAL REVIEW

This section contains the contract and technical review comments describing the findings and observations for each of the main verification and validation parameters described in GDE-205. The actions taken for each analysis and the reasons why a particular data qualifier flag was assigned are also included. The following verification and validation parameters were reviewed.

A. COMPLETENESS OF THE DATA REPORT PACKAGE

The STL data package for SDG# E0730500101RH was not complete and did not meet all the required Standard + Raw Data Deliverable reporting requirements described in ER-SOW-394, as primary calibration information was missing for gamma detectors, and there were no control charts for any of the gamma or strontium detectors. However, these omissions did not result in data qualification (see section 4.N of this report).

B. RADIOANALYTICAL REPORTING ELEMENTS

Results, uncertainties, and MDCs were not reported to two decimal places (i.e. "x.xxE+xx") as required by ER-SOW-394, sec. 6.1.2.1. Also, the results and uncertainties are not always reported with the same number of decimal places, contrary to GDE-205, sec. 4.3.2.10. However, in the judgment of the validator, these are minor deviations, and no data qualification is warranted.

According to the laboratory case narrative, all Ra-226 results may be biased low, as the samples did not have a 21-day ingrowth period. Accordingly, all positive Ra-226 results have been qualified "J."

The Total Sr LCS was not reported on the LCS report form. However, LCS results were obtained from the raw data.

C. SAMPLE PRESERVATION

Data package documentation indicates that all samples were preserved properly for the required/requested analyses.

D. HOLDING TIMES

The holding time requirements for all analyses (<6 months) were met for this SDG.

E. ANALYTICAL YIELDS

A tracer or carrier is used to measure and correct for losses that might have occurred during sample processing, separation, and quantification of the analyte (in a specific sample). Abnormally high or low chemical yields might be indicative of inappropriate separation methods for certain matrix interferences, instrument problems, calibration errors, or errors in the preparation of the tracer or carrier.

All Total Sr analyses exhibited acceptable yields as described in GDE-205, sec. 4.5.3.1.

Analytical yields are not applicable to the other analyses in this SDG.

F. REQUIRED DETECTION LEVEL

The following samples exhibited MDAs higher than the CRDL listed in ER-SOW-394, sec. 6.2.1, Table 6-4. The calculated MDCs were also higher, indicating that the CRDL was not met for these samples. No data qualification is warranted, per GDE-205, sec. 4.5.4.3.

- All non-detect Am-241, Sb-125, Ce-144, Eu-152, Eu-154, Eu-155, Ru-106, Zn-65, and Zr-95 results for all samples and the duplicate.
- Co-60 for sample E0730500102RH.
- Nb-95 for sample E0730500301RH.
- U-235 for sample E0730500701RH and the duplicate.
- Sb-125, Ce-144, Eu-152, Eu-154, Ru-106, U-235, Th-232, Ra-228, and U-238 for the blank.

G. NUCLIDE IDENTIFICATION

Raw spectral data were provided for gamma analyses. Visual inspection supports proper identification of nuclides. Spectral data were not provided for Total Sr, as it is identified by chemical separation/decay.

H. QUANTIFICATION AND COMBINED STANDARD UNCERTAINTY

Calculation verifications demonstrated that results for all analytes were reported correctly (see Attachment 4). No excessive uncertainty was noted.

I. DETECTABILITY

The detectability status of results (per GDE-205, sec. 4.5.7.3) is listed below:

The following results were statistically positive:

- Cs-137 and Ra-226 for all samples (*see section 4.B of this report for further qualification of Ra-226 results*).
- U-235 for samples E0730500101RH, E0730500301RH, E0730500501RH, E0730500601RH, and E0730500801RH.
- Th-232 for samples E0730500101RH, E0730500301RH, E0730500401RH, E0730500601RH, E0730500701RH, and E0730500801RH.
- Ag-110m for sample E0730500301RH.
- Eu-155 for sample E0730500401RH.
- Ra-228 for sample E0730500501RH.
- Total Sr for all samples except E0730500101RH (*see section 4.J of this report for further qualifications*).

See further qualification of U-235 results for samples E0730500301RH, E0730500501RH, E0730500601RH, and E0730500801RH; Ag-110m for sample E0730500301RH; Eu-155 for sample E0730500401RH;

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The following results were assigned "J" qualifications:

- Th-232 for sample E0730500102RH.
- U-238 for sample E0730500201RH.

The following results were assigned "UJ" qualifications:

- U-235 for samples E0730500102RH, E0730500201RH, and E0730500401RH.
- Cs-134 and Eu-155 for sample E0730500301RH.

All other results exhibited no detectable activity, and have been qualified "U."

J. LABORATORY CONTROL SAMPLES

Laboratory Control Samples (LCSs) are used to assess the bias and precision of the analytical process independent of the field samples. The LCS results (percent recovery) are also used to indicate whether the laboratory's radiochemical procedure is capable of recovering the radionuclide of interest.

The Total Sr LCS data was not included on the LCS report form. However, LCS data was obtained from the raw data. Total Sr LCS recovery was 73%, which is less than the 75-125% acceptance criteria. Per GDE-205, sec. 4.6.1.3, all statistically positive Total Sr results (i.e. all except E0730500101RH) have been qualified "J." The Total Sr LCS was spiked at a level within the 5-20X RDL criteria specified in GDE-205, sec. 4.6.1.1.D.

All gamma LCS recoveries were within the 75-125% acceptance range specified in GDE-205, sec. 4.6.1.1.F, and were spiked at levels greater than the 5-20X RDL criteria. No data qualification is warranted.

K. MATRIX SPIKES

A matrix spike (MS) consists of analysis of an actual sample to which a known quantity of the analyte has been added. Recovery (determined as the percentage of "found" analyte relative to the known amount introduced) provides information on sample-specific matrix effects that result in an analytical bias for a given analysis batch. Matrix spikes are added as early in the sample preparation steps as practicable.

The Total Sr matrix spike exhibited recovery within the 60-140% acceptance range, per GDE-205, sec. 4.6.2.1.D. It was spiked at a level within the 5-20X RDL criteria.

Matrix spikes are not applicable to the other analytes in this SDG.

L. BLANK SAMPLES

A batch blank (method blank) is analyzed concurrently with each set of project samples (sample delivery group, SDG). The batch blank is a laboratory-generated sample prepared with absence of the analyte of interest. Batch blanks are batch quality indicators and are carried through the entire sample analysis procedure with the samples in the batch. The blank should be of the same (or similar) matrix as the project samples and should be a means of determining the existence and magnitude of contamination resulting from the sample preparation and analysis/measurement process (such as from reagents, glassware, equipment, instruments, and/or cross contamination between samples). Any targeted radionuclide activity detected in a blank indicates a potential positive bias in the project sample results for that radionuclide. Potential bias is assessed using the Mean Difference (MD) and Difference Factor (DF) tests described in GDE-205, sections 4.6.3.2.5 and 4.6.3.2.6.

Th-232, Ra-228, and U-238 results were reported for the project samples, but not on the blank report form. However, the Th-232, Ra-228, and U-238 blank results were obtained from the raw data, allowing blank results for all reported analytes to be evaluated.

The Ra-226 blank was statistically positive. Qualifications are as follows:

- Samples E0730500201RH and E0730500401RH each exhibited a MD>3 and a DF>10 (see Attachment 4). No qualification is warranted.
- All other results exhibited a MD>3 and a DF<10. They would normally be qualified "J." However, they have already been qualified "J" due to a possible low bias (see section 4.B of this report). The positive blank activity adds further merit to these qualifications.

All other blanks were statistical non-detects.

M. DUPLICATE SAMPLES

A laboratory-generated duplicate (split) is analyzed concurrently with each set of project samples (SDG) for each analysis and reported on the batch QC reporting form(s). Duplicate analyses can indicate analytical variability and laboratory precision, or the homogeneity/heterogeneity of the sample. For a duplicate sample to meet the acceptance criteria outlined in GDE-205, sec. 4.6.4, sample precision must be ≤ 3 for the mean difference (MD) and/or $\leq 20\%$ relative percent difference (RPD) for water samples (30% for soil samples). However, the mean difference takes precedence over the calculation and use of RPD for duplicate precision (GDE-205, sec. 4.6.4.4).

A sample duplicate was not reported for Total Sr. Instead, a matrix spike and matrix spike duplicate were used to evaluate laboratory precision. In the judgment of the validator, no data qualification is warranted.

All duplicate results demonstrated acceptable laboratory precision with either:

(1) MD values <3 for results with statistically positive sample results and statistically positive duplicate results,

(2) MD values <3, using the ½ CRDL method, or

(3) both sample and duplicate results that exhibited non-detectable activity (See Attachment 4).

N. EFFICIENCY, ENERGY, AND BACKGROUND CALIBRATION

The data package did not include primary calibration information for gamma detectors. Also, the data package did not include control charts for the gamma or strontium detectors. However, in the judgment of the validator, no qualification is warranted, as the daily calibration checks were all acceptable.

All other efficiency, energy, and background calibration information indicates that the instruments were operating properly during the counting/analysis of the reported sample results.

O. PERFORMANCE EVALUATION SAMPLE

There were no INL performance evaluation samples noted in the transmittal of this report, nor on any of the official documents. Therefore, no evaluation of INL PE standards was conducted.

5. DATA LIMITATIONS AND USABILITY OVERVIEW

This section provides an overview of the limitations of the data for each sample and for each analysis.

5.1 Summary of Qualified Data

The radionuclide analyses that received data qualifier flags are listed below. The qualifier flags applied to the data for this SDG are presented in Table 5.2. The data qualifier flags used and their definitions are shown in Table 5.3.

Gamma Isotopes

The following results were left unqualified. The radionuclides are considered present in the samples.

- Cs-137 for all samples.
- U-235 for samples E0730500101RH, E0730500301RH, E0730500501RH, E0730500601RH, and E0730500801RH.
- Th-232 for samples E0730500101RH, E0730500301RH, E0730500401RH, E0730500601RH, E0730500701RH, and E0730500801RH.
- Ag-110m for sample E0730500301RH.
- Eu-155 for sample E0730500401RH.
- Ra-228 for sample E0730500501RH.

See further qualification of U-235 results for samples E0730500301RH, E0730500501RH, E0730500601RH, and E0730500801RH; Ag-110m for sample E0730500301RH; Eu-155 for sample E0730500401RH;

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The Ra-226 results for all samples have been qualified "J" due to a possible low bias (see section 4.B of this report). The radionuclide is considered present in the samples, but the values may be estimated.

The following results were qualified "J" per GDE-205, sec. 4.5.7.3. The radionuclides are considered present in the samples, but the values may be estimated.

- Th-232 for sample E0730500102RH.
- U-238 for sample E0730500201RH.

The following results were qualified "UJ" per GDE-205, sec. 4.5.7.3. The radionuclides are not considered truly present in the samples, and the use of the results is strongly discouraged.

- U-235 for samples E0730500102RH, E0730500201RH, and E0730500401RH.
- Cs-134 and Eu-155 for sample E0730500301RH.

All remaining gamma results were statistical non-detects, and have been qualified "U." The radionuclides are not considered present in the samples.

Total Sr

All Total Sr results, except E0730500101RH, were qualified "J" per GDE-205, sec. 4.6.1.3 (see section 4.J of this report). The radionuclide is considered present in the samples, but the values may be estimated.

The Total Sr result for sample E0730500101RH was a statistical non-detect, and has been qualified "U." The radionuclide is not considered present in the sample.

5.2 Radioanalytical Data Qualifier (Validation Flag) Table

	Analysis Type
Sample Number	Total Sr
E0730500101RH	U
E0730500102RH	**J**
E0730500201RH	**J**
E0730500301RH	**J**
E0730500401RH	**J**
E0730500501RH	**J**
E0730500601RH	**J**
E0730500701RH	**J**
E0730500801RH	**J**

	Analysis Type																						
Sample Number	Am-241	Sb-125	Ce-144	Cs-134	Cs-137	Co-58	Co-60	Eu-152	Eu-154	Eu-155	Mn-54	Nb-95	Ra-226	Ru-103	Ru-106	Ag-108m	Ag-110m	U-235	Zn-65	Zr-95	Th-232	U-238	Ra-228
E0730500101RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U		U	U			
E0730500102RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	**UJ**	U	U	**J**		
E0730500201RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	**UJ**	U	U		**J**	
E0730500301RH	U	U	U	**UJ**		U	U	U	U	**UJ**	U	U	**J**	U	U	U	J	J	U	U			
E0730500401RH	U	U	U	U		U	U	U	U	J	U	U	**J**	U	U	U	U	**UJ**	U	U			
E0730500501RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	J	U	U			
E0730500601RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	J	U	U			
E0730500701RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	U	U	U			
E0730500801RH	U	U	U	U		U	U	U	U	U	U	U	**J**	U	U	U	U	J	U	U			

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Table 5.3. Data qualifier flag definitions.

Flag	Definition
<none>	The analysis was performed, and radioactivity was detected (e.g., the radioanalytical result is statistically positive at the 95% confidence interval and is above its MDC). **NOTE:** *The radionuclide is considered to be present in the sample.*
U	The analysis was performed, but no radioactivity was detected (i.e., the radioanalytical result was not statistically positive at the 95% confidence interval and/or the result was below its MDC). The "U" qualifier flag is also applicable to any result reported as zero (0) (± an associated uncertainty). **NOTE:** *The radionuclide is not considered to be present in the sample.*
UJ	The analysis was performed, however, the result is highly questionable due to analytical and/or laboratory quality control anomalies. The use of such a result is strongly discouraged. Analytical and quality control anomalies include such items as: significant blank contamination, known photopeak interferences and/or photopeak resolution problems, known matrix interferences, unacceptable laboratory control sample recoveries, serious instrument calibration problems, improper sample preservation, etc. The "UJ" qualifier flag could designate a possible false positive result in the case of a result that is statistically positive at the 95% confidence level. The "UJ" qualifier flag could indicate the result is considered an estimated nondetect (a nondetect that may be due to loss of analyte from lack of sample preservation, holding time exceedences, etc.). The specific use of the "UJ" flag is included by the validator in the text of the validation report. **NOTE:** *The radionuclide may or may not be present in the sample and the result is considered highly questionable.*
J	The analysis was performed, and radioactivity was detected (i.e., the radionuclide result is statistically positive at the 95% confidence interval and is above its MDC). However, the result is questionable due to analytical and/or laboratory quality control anomalies/irregularities and should therefore be used only as an estimated (approximated) quantity. Analytical and/or quality control anomalies include such items such as: laboratory duplicate imprecision, unsatisfactory analytical yields, insufficient laboratory control sample recoveries, unacceptable PE sample results, instrument calibration problems, improper sample preservation, etc. **NOTE:** *The radionuclide is considered to be present in the sample; however, the result may not be an accurate representation of the amount of activity actually present in the sample.*
R	The analysis result is unusable and was rejected due to severe analytical and/or quality control problems. **NOTE:** *The radionuclide may or may not be present, and the result is known to be inaccurate or imprecise.*

6. DEFINITIONS

Accuracy. Accuracy is the degree of agreement of a measurement with the known (reference) value of a calibrated (certified) standard, source, or reference material.

Achieved Detection Limit (ADL). The minimum amount of sample activity that can be detected or identified with 95% confidence for a particular analysis based on sample-specific analysis and measurement conditions. See definition for detection limits. Further discussion about detection limits can also be found in Section 4.11.

Aliquot. It is a portion of the total sample used in the analysis.

Background. Ambient signal response due to spurious electronic noise or incidental radiation in the vicinity of the detector system as recorded by measuring instruments that is independent of radioactivity contributed by the radionuclides being measured in the sample.

Bias. A positive or negative deviation of the measured value from the assumed or accepted true value which does not tend toward zero.

Blind. QC samples that are prepared external to the analytical laboratory and are submitted to the laboratory unknowingly along with a regular set of samples.

Calibration (efficiency). A method of measuring and establishing the response of an instrument with the use of a calibration standard, sources, or samples. The response is a calibration factor or curve that corrects for the difference between the known number of radiation quanta emitted by a source and the actual number measured/detected by the instrument. Detection systems are only capable of detecting a fraction of the radioactivity actually being emitted from the radioactive nuclides. Therefore, in order to make quantitative determinations, it is imperative to establish the relationship between the measured counting rates and that of the known emission (or disintegration) rates. Such a relationship is commonly referred to as the detector efficiency. The efficiency is typically expressed as a ratio or percentage of the measured counting rate to the known disintegration rate of the radioactive calibration standard/source (Eff. = cpm/dpm). Detector efficiency is the essential element for the quantification of radioactivity in samples.

Calibration (energy). A method of calibrating an instrument for its channels versus energy relationship with the use of radioactive sources of well-known photon or particle energies. Detection systems using multi-channel analyzers (for spectrometric analyses) must be calibrated for the energy of the quanta (alpha particles or gamma rays) being emitted from the radioactive material, in order to make correct radionuclide identification (i.e., qualitative measurements).

Carrier. Carriers are typically non-radioactive (e.g. natural strontium, barium, yttrium) elements. They follow similar chemical reactions as the analyte during processing and are added to samples to determine the overall chemical yield for the analytical preparation steps. The yield of the carrier is typically determined gravimetrically.

Chain-of-Custody (COC). A history of the transfer of samples from the time of sample acquisition to the final disposal of the samples. It provides a tracking mechanism that allows the possession, handling, and security (custody) of individual samples to be maintained and traced from the time of sample collection, through laboratory analyses, to the final disposition.

Combined Standard Uncertainty. The total uncertainty associated with a sample measurement result, and includes all the individual uncertainty components incurred in the entire analytical/measurement process. The CSU is the addition of the square root of the sum of the squares of random components of the individual uncertainties, plus the magnitude of the estimated individual systematic uncertainties (often referred to as propagating the uncertainties in quadrature). For purposes of this guide, CSU includes only those random and systematic uncertainties associated with the analytical process and does not include the uncertainties associated with field sampling. The mathematical expression is as follows:

$$TPU = \sqrt{(\Sigma\sigma^2_{random} + \Sigma\sigma^2_{systematic})}$$

Confidence Interval. A statistical distribution (band or interval) around a measured value within which the "true value" is expected to lie. This interval equates to the degree of confidence we have in the measured value, and is expressed as either a percentage or a standard deviation. Also see definitions for *Precision* and *Uncertainty.*

Data Package. The report received from the laboratory containing the analytical results and supporting documentation for a set of samples. The contents and format of the data package are often specified by the client.

Data Qualifier Flag. The flag (letter codes) assigned to individual sample results during the data validation process to indicate the potential limitations and usability of the sample data. See Table B-2 of GDE-205 for a definition of each qualifier flag.

Detection Limit. The minimum amount of radioactivity that can be reliably detected (with an established degree of confidence) under certain defined sets of background, sample, instrument, analytical, and measurement conditions. It is typically defined (by L.A. Currie, who is one of the foremost, recognized authorities in radiation statistics and detection limits) as $2.71+ 4.65\ (B)^{1/4}$, where B is the background value. The detection limit is usually expressed as a two-sided, probabilistic expression set far enough above zero (0) so that it includes both Type I and Type II errors. The probabilities associated with the Type I error (a 5% chance of deciding radioactivity is not present when it is not - false positive) and the Type II error (a 5% chance of deciding radioactivity is not present when it is - false negative) assure us that the established detection limit (i.e., the detector response level and/or the sample response level) will detect activity in a sample (with 95% confidence), if it is present.

The detection limit is defined/described by various terminologies such as, *Lower Limit of Detection (LLD), Minimum Detectable Activity (MDA), Minimum Detectable Concentration (MDC), Detection Sensitivity, a-priori, a-posteriori, etc.* These terminologies are often mistakenly interchanged, misused, and misapplied. Most are not synonymous and each has a different meaning and specific application. Generally, LLD refers to limits determined from appropriated blanks that are truly representative of the samples being analyzed, MDA and MDC refers to a limit that is sample-specific and is

determined from the actual sample being measured, *a-priori* is a limit that is more synonymous with the LLD and is a "before-the-measurement" estimate of what can be detected under 'ideal' sample and analysis conditions (where the sample and analytical variables can be controlled or held constant), and a-posteriori is a limit that is more synonymous with the MDA and MDC and is an "after-the-measurement" determination of what is actually detectable.

The detection limit often used as a probabilistic and statistical attempt to define and or determine whether radioactive material is present in a sample or not. A more practical definition is best defined by the function the detection limit performs: (1) it establishes a detector response level, which when exceeded, indicates that radioactive material is present in a sample, and (2) it establishes the sample activity necessary so that we can be assured that the radioactive material present in a sample will be detected.

FWHM. Full width at half maximum.

False Positive Result. A false positive result, in the context of GDE-205, means that a statistically positive detection was made, however, the activity was due to blank contamination, photo-peak interferences, or instrumental/ambient background contributions (i.e., no radioactivity is present in the sample).

Laboratory Blank. A laboratory-generated sample, representative of the sample matrix being analyzed, that contains none of the constituents of interest that has gone through the entire analytical and measurement process using the same reagents added to the samples being analyzed. The blank provides verification that contamination has not occurred during the handling, preparation, and analysis of the samples.

Laboratory Control Sample (LCS). The LCS is a certified material or an aliquot of a matrix (blank), which is free of radionuclide interferences (and the constituents of interest), that is spiked with a known concentration of a target radionuclide(s) and is put through the entire analytical/measurement process. Provides an indication of the adequacy of the laboratory procedure to measure the constituent of interest.

Laboratory Duplicate. A laboratory-generated split of an actual sample that is put through the same exact analytical/measurement process as the original sample. Provides an indication of analytical variability/precision or sample inhomogeneity.

Matrix. The media in which a radioactive material of a sample is embedded.

Matrix Spike Sample (MSS). An aliquot or aliquant of a sample spiked with a known concentration of target analyte(s) prior to sample preparation. The recovery of the target analyte(s) from the MSS is used to determine the bias of the method in the specific sample matrix.

Mean Difference (MD). A standard statistical method of assessing differences between radioactivity measurements and determining the significance of those differences. It is used in this procedure to evaluate the statistical difference between method blank results and sample results and to evaluate results associated with duplicate measurements.

Minimum Detectable Activity (MDA). See definition for detection limits.

Minimum Detectable Concentration (MDC). See definition for detection limits.

Non-Detect. A statistical interpretation that indicates the "absence" of radioactivity in a sample when the analytical result is less than two times the reported one sigma error of that result.

Performance Evaluation (PE) Sample. A QC sample that is specifically prepared with a known (reference or traceable) amount of radioactive material and submitted blind to the laboratory with a batch of field samples. It is used under sponsorship of the INL SMO as a real-time tool to assess and monitor the laboratory's proficiency in performing "routine" radioanalytical measurements on samples they believe are regular samples.

Precision. A measure of the variability of a set of repeated measurements of the same quantity using the same analytical technique or instrument. It can also be referred to reproducibility or repeatability. It is typically expressed quantitatively as the standard deviation of the results obtained from the series of measurements.

In radiation measurements usually only a single measurement is made (and repeated measurements are not necessary), because the variability associated with the randomness of the radioactive decay process is very well understood and predictable. This randomness (or variability) follows the basic laws of probabilities and probability distributions, which are described by Poisson and Gaussian statistics. The degree of precision is dependent on the intensity of the radioactivity being measured. Precision is often referred to as an uncertainty and is typically expressed quantitatively as a standard deviation. See the definition of Confidence Interval and Uncertainty.

Quality Assurance Flag. See Data Quality Assessment Flag.

Quality Assurance (QA). A network of activities that assures that all information, data, and decisions are technically sound and properly documented, that the quality objectives are met, and that the results of analyses are correct (within the associated method uncertainties). These activities include evaluating the data quality objectives, designing these into the laboratory requirement documents, monitoring the quality of analytical results by inspection and by the injection of quality control samples, and assuring that the personnel performing the analysis are qualified.

Quality Control (QC). Quality Control is the mechanism by which the QA system is directed. It is a system of activities whose purpose is to monitor and ensure the quality of the analysis and measurement process. This consists of routine performance tests and checks with QC materials (standards, sources, and samples) to verify the accuracy and precision of analysis/measurement process.

Random Error. The deviation or variation of a measured value due to certain characteristics and fluctuations in the measurement process that cannot be controlled (e.g., randomness of the radioactive decay process, sample inhomogeneity, and sample positioning repeatability). It is the random occurrences that result in a change from one measurement to another causing the measured value to be somewhat different from other measured values.

Required detection level (RDL). The minimum detection capability for a method required by the project and/or statement of work.

Sample Delivery Group (SDG). A group or batch of samples from one sampling project, received by the laboratory over no more than a 14-calendar-day period, that does not exceed 20 samples. The group of samples are analyzed together and reported in one data package. The SDG number is assigned by the laboratory and one of the INL sample numbers included in the sample delivery group.

Source (calibration). A radioactive source of a standardized matrix and geometry that is used for detector efficiency calibration or calibration checks. The radioactive source typically contains standard reference materials for which the activity of the radionuclide(s) is traceable, known, and certified within specified limits of uncertainty.

Standard (calibration). A radioactive source that is always used as whole, such that the activity for the whole source is quoted by the supplier and certified by a recognized standardizing agency or group (e.g., NIST).

Standard Reference Material (SRM). Material specially prepared, analyzed, and certified for radioactivity content under the jurisdiction of a recognized standardizing agency or group, such as the National Institute of Standards and Technology (NIST), for use by analytical laboratories as a calibration material or as an accurate basis for comparison.

Statistically Positive. A statistical determination that identifies the "presence" of radioactivity in a sample when the analytical result is greater than two times the reported one sigma error of that result.

Systematic Error. The inherent bias (offset) of a measurement process. It is the errors that are fixed or constant during the time measurements are being made (e.g., efficiency calibration, procedure, instrumentation, and nuclear decay data).

Task Order Statement of Work (TOS). A written work order or statement of analytical requests and requirements for the laboratory, prepared by the SAM, that delineates project specific information such as, numbers of samples to be collected, sample collection schedule, and types of analyses requested. The TOS also describes any deviations, additions, deletions, or changes to the SAM analytical statement of work.

Traceable (standards). All detection systems are calibrated with radionuclide reference materials, sources and standards traceable to accredited/certified national reference laboratories such as the National Institute of Standards and Technology (NIST-USA), the Physikalische Technische Bundesanstalt (PTB-Germany), and the National Physical Laboratory (NPL-United Kingdom). Traceability provides a documented pedigree, confirmation, and assurance of accuracy and precision.

Tracer. A radionuclide that chemically mimics and does not interfere with the target radioanalyte through the chemical preparation and instrument analysis.

Uncertainty. The variability (or inaccuracy) associated with measured value due to the statistical (random) fluctuations in the measurement system or process. It represents the band around the measured value within which the "true value" is expected to lie. It is often expressed as a standard deviation or a percentage, and is always described with an associated confidence level. See definitions for *Confidence Interval, Precision, and Combined Standard Uncertainty.*

Validation. A technically-based analyte- and sample-specific evaluation process that extends beyond method or contractual compliance, provides a level of confidence that an analyte is present or absent, and examines the uncertainty of the reported concentration of the analyte relative to the intended use of the data. Data validation is a systematic process, performed externally from the data generator, that applies a defined set of performance-based criteria to a body of data that can result in qualification of the data. Data validation occurs prior to drawing a conclusion from a body of data.

Validator. An individual trained to GDE-205 and the INL radioanalytical statement of work that performs data validation on analytical report data packages. The individual(s) should have a scientific or technical background in the field of radiochemistry.

Verification. A review and evaluation process used to determine that laboratory operations, data quality elements, and the resultant data are compliant with contractual agreements and requirements.

Yield. Is a measure of the efficiency of the radiochemical separation process. It is determined by adding a known amount of radioactive tracer or chemical carrier to the sample prior to sample preparation and analysis and measuring the analytical yield (gravimetrically or radiometrically) at the completion of the analytical/measurement process. The yield determinations are used in the calculation of sample results.

7. REFERENCES

A. Idaho Cleanup Project, "*Radioanalytical Data Validation*" Sample and Analysis Management, GDE-205, Rev.1, May 2004.

B. Idaho Cleanup Project, "*Sample and Analysis Management Statement of Work for Analytical Services*," ER-SOW-394, Rev.3, March 2005.